Section XII: Identification of Lysergic Acid Diethylamide (LSD)

I. Introduction:

LSD samples are analyzed by GC/MS. When sample amount permits, screen samples by Thin Layer Chromatography (TLC).

II. Reagents:

A. Ethanol
B. Methanol
C. Ammonium Hydroxide (NH_4OH)
D. LSD Standard

III. Equipment:

A. Glass microvials.
B. 2 mL autosampler vials and Teflon caps.
C. Tweezers
D. Glass cutter
E. 10 uL autosampler syringe
F. 100 mL graduated cylinder
G. Glass evaporation beaker
H. TLC Plate ($CaCO_3$)
I. TLC Chamber
J. Model CC-80 UV Fluorescence Analysis Cabinet
K. GC/MS: HP 5890/5972 series.

IV. Procedure:

A. Screening by Thin Layer Chromatography (TLC):

1. Prepare TLC chamber by making a solution of NH_4OH and Methanol. Add 1.5 mL of NH_4OH into a 100 mL graduated cylinder and bring to volume with Methanol. Add solution to the TLC Chamber.
2. Place the sample into a glass evaporation beaker. Add 1-3 drops of Methanol or Ethanol.
3. Label the columns on the TLC plate, two standard runs and sample numbers.
4. With a glass cutter, score the plate at the $CaCO_3$ end where the standard and samples are to be added.
5. Clean and rinse syringe with Methanol. Be sure to rinse between each sample also.

6. Add about 1 uL/drop of each standard to the appropriate columns of the TLC plate.
7. Add about 1 uL/drop of each sample to the appropriate columns on the TLC plate.
8. Measure 120 mm from the end of the $CaCO_3$ and mark it with a pencil.
9. Insert the TLC plate into the TLC chamber, cover and allow to elute for about 45-50 minutes or until the solvent reaches the 120 mm pencil mark.
10. Take the plate out of the chamber and mark the line where the solvent stopped with a pencil.
11. Allow plate to dry for 10-20 minutes.
12. When dry, place the plate in the UV Analysis Cabinet
13. Turn on the cabinet and long wavelength (365nm).
14. Read the plate. With a pencil, draw a circle around the LSD standard mark, as well as for the samples.
15. Measure the distance traveled by the LSD. Measure using the center of the spot.
16. Report Rf values for each substance. The Rf value is defined as:

$$Rf = \frac{\text{Distance substance traveled from origin}}{\text{Distance solvent front travels from origin}}$$

17. If sample results are positive, then proceed to Step B, Direct Injection.

B. Direct Injection on GC/MS:

1. This method is done by direct injection on the GC/MS, therefore the standard and blank should be analyzed first before the sample is prepped. However, if the screening procedure was performed then the sample is already prepared so proceed to next step.
2. Direct inject about 1.5 uL of standard. Then direct inject 1 uL of blank. If the sample still needs to be prepped, place the sample in a microvial inserted into an autosampler vial and place 1-3 drops of Ethanol into it, then cap.
3. Direct inject 5-7 uL of the sample.
4. When run is completed and report generated, remove sample (if in paper form) from microvial and return to evidence bag.
5. The GC/FID conditions are:
   Method: LSD.M
   Oven:
   Initial Temp: 55°C
   Initial Time: 0.50 min.
   Max. Temp: 300°C
   Rate: 60 °/min.
   Inlet: (front injector only)
   Mode: Split
   Initial Temp: 250 °C

Pressure: 30.0 psi
Gas Type: Helium

Column:

Capillary: 5972: HP-5MS 25m x 200um x 0.33um
5973: HP-1MS 25m x 200um x 0.33um

6. If LSD is present in the sample the instrument will detect a peak at approximately 14.26 minutes on the 5890/5972 and at approximately 13.00 minutes on the 6890/5973. A report will be generates along with accompanying chromatograph and spectra. The spectra will contain of the peak and its ion abundance (See graph, last graph).

V. Results:

A. Record results of GC/MS in logbook and transfer the results to appropriate evidence cards, as well as any paperwork that came with the sample. Be sure to include the date of analysis, result, and initials on the evidence cards.
B. All reports generated from the instrument should be filed so they may be accessed at a later date, if necessary